





Metadata in high pressure crystallography

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Challenge: consistent metadata descriptors

- The main metadata source in high pressure crystallography is the result of maintaining the sample in the pressure-transmitting environment
- The components of the cell interfere with the primary and diffracted beam, what contaminates the diffraction patterns and affect reflection intensities
- Different sample environments (diamond anvil cells, Paris-Edinburg cells, multi-anvil presses, tube furnaces etc.) implies various protocols for data treatment and different metadata items
- <u>Always be critical</u> of the HP diffraction data!

Diamond anvil cell (opposed anvil assembly)



The principle of pressure generation



The principle of pressure generation



The principle of pressure generation





Access in reciprocal space





toroidal shape of accessible part of the reciprocal space

Access in reciprocal space



accessible reciprocal space depends on the cell opening angle!

Limitations in reciprocal space



Limitations in reciprocal space



Anvils and backing-seat design



Corrections for beam-shadowing effects



stages of *in situ* crystallization from the melt (ethynylbenzene)

NOTA BENE:

- if the crystal fills partially the pressure chamber, the microphotograph of the sample could be useful!
- if the beam waist is much smaller (e.g. synchrotron radiation), no gasket shadowing correction is needed







A. Katrusiak, Z. Kristallogr. 2004, 219, 461-467.

Computer programs: REDSHABS (A. Katrusiak), ABSORB (R. Angel)

Most essential metadata in high-pressure crystallography (X-ray diffraction; DAC)

- Orientation of the DAC with respect to the incident beam and the detector
- Sample shape (single crystal, powder), sample preparation and history
- Dimensions of the DAC components (anvil thickness, gasket thickness, chamber diameter, backing-plate type, aperture opening angle, etc.)
- Chemical composition of the cell parts (anvils, gasket, backing plates) and their absorption coefficients
- Pressure-transmitting medium
- Sample photo
- Other types of pressure cells and techniques (e.g. neutron diffraction) require different metadata sets

HP metadata as descriptors in CIF

• CoreCIF: '_diffrn_measurement_specimen_suport' (e.g. 'diamond anvil cell'),

- '_diffrn_ambient_environment' (definition: *the gas or liquid surrounding the sample, if not air*; can describe pressure-transmitting medium),
- '_diffrn_ambient_pressure' (the mean hydrostatic pressure in kilopascals at which the intensities were measured),
- '_diffrn_ambient_pressure_gt', '_diffrn_ambient_pressure_lt' (pressure range),
- '_cell_measurement_pressure' (the pressure in kilopascals at which the unit-cell parameters were measured),

'_exptl_crystal_pressure_history' (relevant details concerning the pressure history of the sample),

'_exptl_crystal_recrystallization_method' (Describes the method used to recrystallize the sample. Sufficient details should be given for the procedure to be repeated. The temperature or temperatures should be given as well as details of the solvent, flux or carrier gas with concentrations or pressures and ambient atmosphere).

HP metadata as descriptors in CIF

- mmCIF: '_diffrn.ambient_pressure_esd' (standard uncertainty),
 - '_cell_measurement.pressure_esd' (standard uncertainty),
 - '_exptl_crystal_grow.pressure' (the ambient pressure in kilopascals at which the crystal

was grown),

'_exptl_crystal_grow.pressure_esd' (standard uncertainty).

- •pdCIF: **'_pd_prep_pressure'** (preparation pressure of the sample in kilopascals. This is particularly important for materials which are metastable at the measurement pressure, _diffrn_ambient_pressure).
- •msCIF: '_cell_wave_vectors_pressure_max', '_cell_wave_vectors_pressure_min' [the maximum and minimum values of the pressure in kilopascals defining the interval within which the modulation wave vector(s) were measured].

HP metadata as descriptors in CIF

• Many of the required metadata items do not yet exist in the current CIF dictionaries as explicit numerical or textual descriptors, but they can be added!

Thank you!

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